

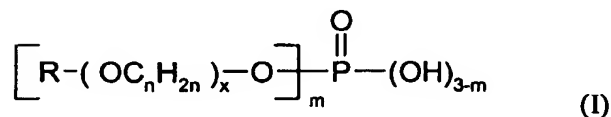
LIQUID TEXTILE-PRETREATING AGENT

The present invention concerns a liquid textile-pretreating agent useful in all continuous
 5 and batch pretreatment operations which is based on phosphoric esters of alkoxyated
 Guerbet alcohols and is very stable to alkali not only in the formulation but also in the
 liquor.

In grey cloth conversion, the pretreatment of the natural or synthetic fibre materials
 10 constitutes an important basis for the further processing. The various operations such as
 desizing, scouring, bleaching or mercerizing employ a wide range of textile chemicals,
 examples being surfactants, dispersants, emulsifiers, bleaches, foam suppressants or
 defoamers. Among the requirements which these auxiliaries as they are known have to
 15 meet is high stability to alkali, especially in the alkaline scouring of woven cotton
 fabric. There is accordingly a constant need for new active compounds having a suitable
 performance profile.

It has now been found that, surprisingly, phosphoric esters of specific alkoxyated
 Guerbet alcohols are very useful for continuous and batch pretreatment of textile
 20 material. Alkoxyated Guerbet alcohols as such are known for example from
 WO 03/091192 A1. Although the use in formulations for the textile industry is
 disclosed there, no pointer is given to the excellent and surprising properties of the
 phosphoric esters.

25 The invention accordingly provides compounds of the formula (I)

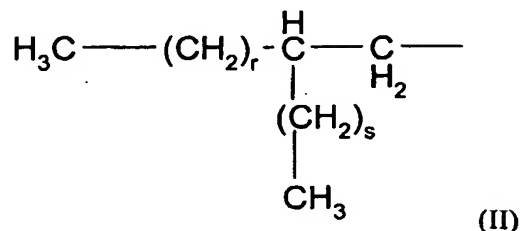


where

30 m is from 1 to 3,
 n is from 2 to 4,

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- x is from 4 to 12, and
 R is a radical of the formula (II)



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where

- r is from 0 to 8,
 s is from 0 to 8,
 the sum total of (r + s) is from 4 to 8, and
 10 the alkyl chains may in turn be linear or branched.

- The present compounds are highly stable surfactants which can be present as a highly concentrated, for example 60% solution in water without any need for additives. This combines with good wettability and low foam-forming tendency into a unique
 15 performance profile. Especially the extremely high stability to alkali not only in the formulation but also in the liquor predestines these compounds for the alkaline scour, but use as a dispersant, as an emulsifier or as a defoamer component is also possible.

Also of very high suitability are compounds wherein

- 20 m is from 1 to 3,
 n is 2 or 3,
 x is from 6 to 8,
 r is from 2 to 6,
 s is from 0 to 4, and
 25 the sum total of (r + s) is from 5 to 7.

Especially good properties are exhibited by compounds wherein

- m is from 1 to 2,
 n is 2,

x is 7,
r is 4,
s is 2, and
the sum total of (r + s) is 6.

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The index m is an average value, and a particularly preferred value for m is from 1.2 to 1.3.

10 The present compounds are prepared by alkoxylation of appropriate Guerbet alcohols as described in WO 03/091192 A1 and subsequent phosphorylation, preferably with phosphorus pentoxide. The alkylene oxide units of the Guerbet alcohols used are mostly ethylene oxide or propylene oxide units, but mainly ethylene oxide units combined with low fractions of propylene oxide or else only ethylene oxide units.

15 Phosphorylation is effected by portionwise addition of the phosphorylating agent at 90 to 120°C during 4 to 24 hours in the absence of air.

The present compounds can be used as such or in the form of an aqueous composition for pretreating textiles.

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The present invention thus further provides a composition comprising an aqueous solution of one or more compounds of the formula (I) and also further auxiliaries.

25 Preferably, the aqueous composition comprises 40% to 70% by weight of compound (I), although about 2% to 4% by weight can also be present in the form of the sodium salts. The composition may further comprise 0.1% to 3.5% by weight of further auxiliary materials, examples being surfactants, biocides, defoamers or foam suppressants.

30 The composition according to the invention is obtainable by simply mixing its constituents.

A preferred use of compounds of the formula (I) or of the abovementioned aqueous composition is the pretreatment of textiles in continuous or batch operations under alkaline conditions.

- 5 The examples which follow illustrate the invention.

EXAMPLES

- 10 There now follows a description of the alkoxyated Guerbet alcohols used, of the products obtained therefrom by phosphation, the aqueous formulations produced therefrom and the resulting applicatory results in table form.

The following test methods were employed:

15 • **Alkali stability**

- What is tested is the alkali stability of 5 g/l of surfactant, with 100 ml of liquor being made up in each case. The test takes place at room temperature 20 to 25°C. The required amount of aqueous sodium hydroxide solution is weighed into a glass beaker and made up to 95 ml with demineralized water. 5 ml of a 10% surfactant solution are added to the alkali batches with stirring. The glass beakers are left to stand at room temperature for 24 hours without stirring.

- 25 The solutions are tested for their stability after 24 hours. Creaming and precipitates are to be noted in particular, cloudiness without visible deposits being permissible. What is to be ascertained is the concentration at which the surfactant is still stable. Alkali stability is reported in X of °Bé- NaOH.

• **Ross-Miles foam test**

- 30 The foam volume is measured after a certain amount of liquid has been poured from a certain height, instantly and after a one minute wait.

A 1000 ml graduated cylinder 60 mm in internal diameter and 430 mm in internal height is used. The test liquid is allowed to pour out from a 2 l separating funnel

through a capillary 70 mm in length and 2 mm in internal diameter from a height of 600 mm, measured from the outlet of the capillary above the floor of the cylinder.

5 500 ml of the solution to be tested are filled into the separating funnel and allowed to flow out into the graduated cylinder through the capillary-controlled efflux rate of about 0.17 l/min. As soon as the entire solution has flowed out, a stopwatch is started and the entire volume (foam volume plus solution volume) is read off the cylinder scale. The reading is repeated after one minute.

10 The alkaline foam performance is tested using a surfactant concentration of 2 g/l in 2° Bé NaOH solution in demineralized water, with 2° Bé NaOH being equivalent to 12 g/l of NaOH solid or 30 ml/l of NaOH 36° Bé. The test temperature is 20 to 25°C.

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- **Alkaline wetting**

This test method determines the number of seconds a fabric sample takes to sink to the bottom of a glass beaker 1 l in content, 14 cm in height and 10 cm in diameter in a surfactant solution. The fabric sample used is a cotton test cloth, from EMPA Testmaterialien AG of St. Gallen, Switzerland. Circularly round discs 3.5 cm in diameter are die cut out of this cloth and dipped with a special holder into the surfactant solution. The wetting action is tested in 2° Bé NaOH at 25°C.

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TABLE 1

Utilized alkoxylated Guerbet alcohols and other alcohols

No.	Alcohol	EO	PO	Residual alcohol
1	not less than 70% of 2-propyl-1-heptanol, not more than 30% of 2-propylisoheptanol	7	0	6
2	not less than 70% of 2-propyl-1-heptanol, not more than 30% of 2-propylisoheptanol	10	0	2
3	not less than 70% of 2-propyl-1-heptanol, not more than 30% of 2-propylisoheptanol	7	1	≤ 1
V1	i-Undecanol (Standard)	7	0	low
V2	Exxal-C11 (i-C11 from Exxon)	7	0	low
V3	i-C12/C14	7	0	low

5 EO, PO = ethylene oxide, propylene oxide; V1 to V3 are comparative alcohols.

TABLE 2

Phosphation

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Alcohol No.		% P4O10	Temperature	No. phosphate
	Molar ratio	Molar ratio	Phosphation in °C	
1	1	1.25	6 h 100°C	I
1	1	1.25	24 h 115°C	II
2	1	1.25	6 h 100°C	III
3	1	1.25	6 h 100°C	IV
3a	0.9 of alcohol 3 + 0.1 of isodecanol	1.25	6 h 100°C	V

COMPARATIVE TESTS				
V 1	1	1.25	6 h 100 °C	V I
V 2	1	1.25	6 h 100 °C	V II
V 3	1	1.25	6 h 100 °C	V III

TABLE 3**5 Aqueous formulations**

Formulation No.	Sub-number	Components Designation	Concentration in % by weight	Comment
I	a	Phosphate I	60.00%	- problem-free formulation - no solubilizer - no foam suppressant
		Caustic soda	12.80%	- no defoamer
		Biocide (Actacid MBS)	0.10%	
		Water	27.10%	
			100.00%	
I	b	Phosphate I	60.00%	- problem-free formulation
		SagTex DSA	0.50%	- no solubilizer
		Caustic soda	12.80%	
		Biocide (Actacid MBS)	0.10%	

		Water	26.60%	
			100.00%	
I	c	Phosphate I	60.00%	- problem-free formulation - no solubilizer
		ZJ 834 (isononanoamide)	3.00%	- no defoamer
		Caustic soda	12.80%	- just foam suppressant (Si-free)
		Biocide (Actacid MBS)	0.10%	
		Water	24.10%	
			100.00%	
I	d	Phosphate I	60.00%	- problem-free formulation
		SagTex DSA	0.20%	- no solubilizer
		ZJ 834 (isononanoamide)	1.00%	
		Caustic soda	12.80%	
		Biocide (Actacid MBS)	0.10%	
		Water	25.90%	
			100.00%	
II	a	Phosphate II (24 h 115°C)	60.00%	- similar to Ib, except for 24 h
		SagTex DSA	0.50%	- phosphated at 115°C - slightly higher viscosity

		Caustic soda	12.80%	- otherwise the same as Ib
		Biocide (Acticid MBS)	0.10%	
		Water	26.60%	
			100.00%	
III	a	Phosphate III	60.00%	- viscous liquid
		SagTex DSA	0.50%	
		Caustic soda	12.20%	
		Biocide (Acticid MBS)	0.10%	
		Water	27.20%	
			100.00%	
III	b	Phosphate III	60.00%	- viscous liquid
		ZJ 834 (isononanoamide)	3.00%	
		Caustic soda	12.20%	
		Biocide (Acticid MBS)	0.10%	
		Water	24.70%	
			100.00%	
IV	a	Phosphate IV	60.00%	- colourless to yellow liquid
		SagTex DSA	1.00%	- may occasionally also be pasty depending on phosphation
		Caustic soda	7.50%	- increased defoamer content
		Biocide (Acticid MBS)	0.00%	

		Water	31.50%	
			100.00%	
V	a	Phosphate V	60.00%	- colourless liquid
		SagTex DSA	1.00%	- like IV, but an additional 10% of isodecanol fraction in phosphated alcohol
		Caustic soda	7.50%	- reduced alkali stability compared with IV, hence not tested any further
		Biocide (Acticid MBS)	0.00%	
		Water	31.50%	
			100.00%	
<u>COMPARATIVE FORMULATIONS</u>				
VI	a	Phosphate V I	60.00%	- brown paste
		SagTex DSA	0.50%	
		Caustic soda	12.20%	
		Biocide (Acticid MBS)	0.10%	
		Water	27.20%	
			100.00%	
VI	b	Phosphate V I	60.00%	- brown paste
		ZJ 834 (isononanoamide)	3.00%	
		Caustic soda	12.20%	

		Biocide (Acticid MBS)	0.10%	
		Water	24.70%	
			100.00%	
VII	a	Phosphate V II	60.00%	- brown paste
		SagTex DSA	0.50%	
		Caustic soda	12.20%	
		Biocide (Acticid MBS)	0.10%	
		Water	27.20%	
			100.00%	
VII	b	Phosphate V II	60.00%	- brown paste
		ZJ 834 (isononanoamide)	3.00%	
		Caustic soda	12.20%	
		Biocide (Acticid MBS)	0.10%	
		Water	24.70%	
			100.00%	
VIII	a	Phosphate V III	30.00%	- brown paste
		SagTex DSA	0.25%	- not stirrable as 60% version and hence not formulatable
		Caustic soda	6.10%	- half concentration
		Biocide (Acticid MBS)	0.05%	
		Water	63.60%	
			100.00%	

VIII	b	Phosphate V III	30.00%	- brown paste
				- not stirrable as 60% version and hence not formulatable
		ZJ 834 (isononanoamide)	1.50%	
		Caustic soda	6.10%	- half concentration
		Biocide (Acticid MBS)	0.05%	
		Water	62.35%	
			100.00%	

TABLE 4

Applicatory results

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Phos- phate No.	Formulation*	Aspect	Visco- sity	Alkali stability	Ross-Miles foam, alkaline (2° Bé)		Wetting, alkaline
					direct	1 min still	
			cPs	° Bé	ml	ml	s
Ia		clear, colourless	225	16	175	90	46
Ib		min. cloudy, colourless	225	16	0	0	42
Ic		clear, colourless	215	16	65	10	40
Id		min. cloudy, colourless	220	16	40	0	41
IIa		moderately viscous	470	16	0	0	50
IIIa		stable, viscous	960	>17	20	0	78
IIIb		stable, viscous	800	>17	60	10	78

IVa	1% of defoamer	stable, liquid	n.b.	15	50	10	50
Va	1% of defoamer	stable, liquid	n.b.	10	n.b.	n.b.	n.b.
COMPARATIVE TESTS							
VIa		brown paste	1700	17	10	0	85
VIb		brown paste	1700	17	40	20	79
VIIa		brown paste	1500	15	50	5	60
VIIb		brown paste	1400	17	20	0	59
VIIIa	30% of AS, twice the amount applied	brown paste	highly viscous	>17	20	0	ca. 200
VIIIb	30% of AS, twice the amount applied	brown paste	highly viscous	>17	80	50	ca. 200

* unless explicitly mentioned, all formations contain 60% active and 0% solubilizer,
and the cloud point is > 80°C for all (applicatory advantage)

n.b. not assessed;

5 AS active substance

The examples show distinctly that the present invention's formulations of the novel phosphoric esters of certain Guerbet alcohols have a very good property profile, i.e. high stability to alkali, minimal foaming and good wetting.